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(54) ULTRA-HIGH PURITY NITROGEN AND OXYGEN GENERATOR AND PROCESS

EXTREM REINER STICKSTOFF- UND SAUERSTOFFGENERATOR UND VERFAHREN

GENERATEUR D'AZOTE ET D'OXYGENE A DEGRE DE PURETE TRES ELEVE ET PROCEDE

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EP 0 593 703 B2

Description

[0001] The present invention relates to a process and to a generator (air separation unit) for the production of ultra-high purity nitrogen which are suitable for use in a semiconductor manufacturing factory or the like, by which ultra-high purity oxygen necessary for the manufacture of semiconductors or other purposes can be produced at the same time.

[0002] To generate ultra-high purity nitrogen a single air rectification column has been used as disclosed in Japanese Utility Model Application Laid-open N° 45,290/1989. If ultra-high purity oxygen is to be produced (with a purity of 99.9999 %) however, a sufficiently high purity of oxygen cannot be obtained, even if a general air rectifying method and purifying method such as adsorption are combined.

[0003] Accordingly, other methods have been used such as electrolysis, which is high in cost.

[0004] One of the inventors has therefore proposed a method as disclosed in Japanese Patent Application Laid-open N° 282,683/1990, in which ultra-high purity oxygen is produced by using, as a feed material, liquid oxygen having a purity as high as 99.0-99.6 %, produced by another air liquefaction-separation unit, and purifying this feed material through rectification.

[0005] However, if according to such methods ultra-high purity nitrogen and ultra-high purity oxygen are directly fed to a semiconductor manufacturing factory through pipelines, it is necessary to install two units for nitrogen and oxygen.

[0006] To the oxygen unit, furthermore, liquid oxygen must be transported from another oxygen generating factory as a feed material.

[0007] The operation of these two units makes an economically large load, including a personnel expense, running cost and maintenance expense. Disadvantageously, the periodical supplement of liquid oxygen to the oxygen unit from another place requires not only a transportation cost but also a storage tank.

[0008] It is known from EP-A-0.229.364 to provide a four column process and generator for the production of ultra-high purity oxygen and nitrogen as described in the pre-characterizing portion of the independent claims of this invention. This system necessarily produces argon, which is separated from the oxygen in the third of the four columns.

[0009] US-A-4977746 describes a four column process for the production of ultra-high purity oxygen. Nitrogen of unspecified purity is also produced. The top gas of the first column is condensed using the bottom liquid of the second column.

[0010] The present invention is intended to solve various disadvantages in the prior art such as those mentioned above and to provide both the products of ultra-high purity nitrogen and ultra-high purity oxygen preferably in the forms of liquid and gas.

[0011] According to the invention, there is provided a process for the production of ultra-high purity nitrogen and oxygen as claimed in Claim 1.

[0012] According to a further aspect of the invention, there is provided an ultra-high purity nitrogen and oxygen generator as claimed in Claim 8.

[0013] In the generator according to the present invention mentioned above, cooled and liquefied compressed feed air is rectified in the rectifying portion of the first rectification column at first so that an ultra-high purity nitrogen product is separated to the upper portion thereof and oxygen-enriched liquid air to the lower portion thereof, respectively, a portion of the oxygen-enriched liquid air is introduced into a second rectification column so that through its rectification, waste gas containing a large amount of nitrogen gas is separated to the top portion thereof and liquid nitrogen to the bottom portion thereof, respectively, and this oxygen is heated so as to be evaporated by a reboiler of the second rectification column.

[0014] The evaporated oxygen is introduced into a third rectification column, so that through its rectification high purity oxygen gas is separated to above the rectifying portion thereof, and liquid oxygen to be returned to the second rectification column, which contains a trace amount of components having higher boiling points than that of oxygen such as hydrocarbons, krypton, xenon, carbon dioxide and moisture, to below the same rectifying portion, respectively.

[0015] The aforementioned high purity oxygen gas is introduced into a fourth rectification column so that through its rectification, a trace amount of components having lower boiling points than that of oxygen such as nitrogen, carbon monoxide and argon are separated to the top portion thereof and ultra-high purity liquid oxygen to the lower liquid reservoir thereof, respectively. This ultra-high purity liquid oxygen will be taken out as a product as it is in the liquid condition, or in the gaseous condition after heating.

[0016] Referring to the accompanying drawing, one embodiment of the ultra-high purity nitrogen and oxygen generator according to the present invention will be described below.

[0017] All the pressures mentioned below represent gauge pressures.

[0018] As shown in Figure 1, feed air, from which dust has been removed by a filter, is compressed to about 8.7 kg/cm^2 by a compressor 1, and subjected to removal of carbon monoxide, hydrogen, moisture and carbon dioxide by means of a carbon monoxide & hydrogen convector and cooling, decarbonating and drying unit 2. Then, the major portion of the feed air is introduced at a temperature of about 20°C through a pipe P2 into a heat exchanger 3, where it is cooled down to about -166°C through a counter current indirect heat exchange with an ultra-high purity nitrogen gas product, a high purity oxygen gas product, oxygen-enriched air and the other waste gas, which will be mentioned hereinafter, and a portion thereof is liquefied, taken out through

a pipe P3, and introduced to the lower portion of a first rectification column 4.

[0019] In the first rectification column 4, nitrogen gas separated to the top portion thereof through the rectification of the feed air in the rectifying portions 4b, 4c, and 4d thereof is introduced to a nitrogen condenser 8 via a pipe P4, where it is liquefied through an indirect heat exchange with oxygen-enriched liquid air, mentioned below, thereby providing high purity liquid nitrogen, and a non-condensed gas containing impurities having lower boiling points than that of nitrogen such a helium and neon is exhausted through a pipe P34. On the other hand, the major portion of the aforesaid liquid nitrogen is returned to a liquid reservoir 4R1 provided in the upper portion of the first rectification column 4 through a pipe P5.

[0020] From the column bottom of the first rectification column 4, oxygen-enriched liquid air (about -172°C) is taken out through a pipe P6, and reduced in pressure to about 4.2 kg/cm^2 by means of an expansion valve V1. Then, a portion of the oxygen-enriched liquid air reduced in pressure is introduced into the aforesaid nitrogen condenser 8 as a cold source. The oxygen-enriched liquid air evaporated in the nitrogen condenser 8 is turned to oxygen-enriched air of about -172°C and taken out thereof through a pipe P7, and it cools down the feed air in the aforementioned heat exchanger 3 so at to be warmed to about -150°C.

[0021] Then, the warmed oxygen-enriched air is taken out of the middle portion of the heat exchange 3 through a pipe P8.

[0022] The cold gas taken out of the heat exchanger 3 is added to a cold gas coming from a pipe P36, which will be mentioned hereinafter, and both the cold gases are fed to an expansion turbine 9, where they are expanded down to about 0.3 kg/cm^2 so as to have a temperature of about -180°C.

[0023] After the expanded gas is removed therefrom through a pipe P9, it is added to a cold gas from a pipe P16, mentioned below, and both the cold gases are introduced to the heat exchanger 3 again, where they are used to cool down the feed air so as to be warmed to normal temperatures, and are removed through a pipe 10. The major portion of this removed gas is directly exhausted to the open air as waste gas, and a portion thereof is sent to the cooling, decarbonating and drying unit 2 via a pipe 11 as a regenerating gas, and then exhausted to the open air.

[0024] The high purity liquid nitrogen returned to the liquid reservoir 4R1 provided in the upper portion of the aforesaid first rectification column 4 is rectified while it flows down in the rectifying portion 4d thereof. As a result, the high purity liquid nitrogen is turned to ultra-high purity liquid nitrogen free from boiling point components, and it is taken out of a liquid reservoir 4R2 through a pipe P12. After the taken-out ultra-high purity liquid nitrogen is reduced in pressure to 7.5 kg/cm^2 by means of an expansion valve V2 and its temperature is further lowered, it is sent to the aforementioned nitrogen condenser 8.

[0025] The ultra-high purity liquid nitrogen which has been used together with the said oxygen-enriched liquid air as a cold source in the nitrogen condenser 8, thereby cooling down and liquefying the aforesaid nitrogen gas, is evaporated by itself, taken out of the nitrogen condenser 8 through a pipe P13 so as to be sent to the heat exchanger 3. The evaporated liquid nitrogen sent to the heat exchanger 3 is warmed to normal temperatures while it cools down the feed air, and taken out thereof through a pipe P14 as an ultra-high purity nitrogen gas product. In addition, a liquid taken out of the liquid reservoir 4R2 through a pipe 33 will be utilized as an ultra-high purity liquid nitrogen product.

[0026] Although the oxygen-enriched liquid air taken out of the column bottom of the first rectification column 4 through the pipe P6 is expanded down to about 4.2 kg/cm^2 by means of the expansion valve V1, and sent to the nitrogen condenser 8, as mentioned above, the remaining part thereof is branched to a pipe P 15, reduced in pressure to about 0.5 kg/cm^2 by means of an expansion valve V3, and then introduced to the upper portion of a second rectification column 5. This oxygen-enriched liquid air is rectified while it flows down in the rectifying portion 5b of the second rectification column 5. As a result, nitrogen and other components having lower boiling points than that of nitrogen are separated therefrom as non-condensed gas, exhausted out of the top portion of the second rectification column 5 through a pipe P16. The exhausted non-condensed gas is reduced in pressure to 0.3 kg/cm^2 by means of an expansion valve V4, and joined to a discharge pipe P9 of the aforementioned expansion turbine 9.

[0027] The liquid oxygen which has rectified while it flows down in the rectifying portion 5b of the second rectification column 5 and stored in the bottom portion thereof, is warmed so as to be partially evaporated by a gas taken out between the rectifying portions 4b and 4c of the first rectification column 4 through a pipe P17 and introduced into a reboiler 5a disposed in the bottom portion of the second rectification column 5 through a valve 5. The evaporated liquid oxygen is then rectified while it rises in the rectifying portion 5b thereof. On the other hand, the gas introduced into the reboiler 5a is liquefied and then returned to the first rectification column 4 at a position below the aforementioned take-out pipe P17 thereof via a pipe P18.

[0028] Between the liquid oxygen reservoir provided in the column bottom of the second rectification column 5 and the rectifying portion 5b thereof, oxygen gas is taken out through a pipe P19, and it is introduced to below the rectifying portion 6b of a third rectification column 6. This oxygen gas is rectified while it rises in the rectifying portion 6b. On the other hand, a portion of the aforesaid high purity liquid nitrogen taken out of the nitrogen condenser 8 through the pipe 5 is branched to a pipe P21, reduced in pressure by means of an expan-

sion valve V6, and then sent to a condenser 6e provided in the top portion of the third rectification column 6 as a cold source through a pipe P22.

[0029] This liquid nitrogen sent to the condenser 6e condenses and liquefies high purity oxygen gas rising in the rectifying portion 6b, so that it is caused to flow down as reflux liquid.

[0030] Owing to the aforementioned rectification, the liquid oxygen containing a slight amount of impurities having higher boiling points than that of oxygen remains in the bottom portion of the third rectification column 6, and it is taken out through a pipe P20 and returned to below the aforesaid take-out pipe P19 of the second rectification column 5. On the other hand, the high purity liquid nitrogen used as a cold source for the top condenser 6e is evaporated and taken out through a pipe P23, and the taken-out liquid nitrogen is reduced in pressure to about 0.3 kg/cm^2 by means of an expansion valve V7, and then exhausted to a waste gas pipe P16.

[0031] From the third rectification column 6 between the rectifying portion 6b and top condenser 6e thereof, high purity oxygen gas free from impurities having higher boiling points than that of oxygen is taken out through a pipe 24, and introduced to the center portion of a fourth rectification column 7, this is a position between the rectifying portions 7b and 7c thereof. This high purity oxygen gas is rectified while it rises in the rectifying portion 7c. As a result, oxygen is liquefied by a top condenser 7e, mentioned below, and a trace amount of impurities having lower boiling points than that of oxygen are taken out of the column top of the fourth rectification column 7 as non-condensed gas through a pipe P26, reduced in pressure to about 0.3 kg/cm2 by means of an expansion valve V10, and then exhausted into the waste gas pipe P16.

[0032] The high purity liquid oxygen liquefied in the top condenser 7e is rectified while it flows down in the rectifying portions 7c and 7b as a reflux liquid to the rectifying portions 7c and 7b, so that it is fumed to ultra-high purity liquid oxygen free from impurities having lower boiling points than that of oxygen, and stored in the column bottom of the fourth rectification column 7 below the rectifying portion 7b thereof. In the liquid reservoir provided in the column bottom of the fourth rectification column 7 is disposed a reboiler 7a, mentioned below, through which a warming gas passes. By means of the reboiler 7a, the ultra-high purity liquid oxygen is warmed so as to be partially evaporated. Then, the evaporated gas is rectified while it rises in the rectifying portions 7b and 7c.

[0033] For a cold source necessary in the top condenser 7e of the fourth rectification column 7, the high purity liquid nitrogen introduced thereto from the pipe P21 via the expansion valve V8 and the pipe P25 is used similarly in the top condenser 6e of the third rectification column 6. This liquid nitrogen is evaporated by itself and taken out through a pipe 27, regulated in pressure by means of an expansion valve V9, and then exhausted into the waste gas pipe P16. On the other hand, the warming gas fed to the reboiler 7a provided in the column bottom is gas which is taken out of the first rectification column 4 between the rectifying portions 4b and 4c thereof through the pipe 17, similarly to the warming gas for the reboiler 5a of the second rectification column 5, branched to a pipe P28, and introduced into the same reboiler 7a via a valve V11. This warming gas itself is then liquefied here and returned to the first rectification column 4 at a position below the aforementioned take-out pipe P17 thereof through a pipe P29.

[0034] The ultra-high purity liquid oxygen stored in the column bottom of the fourth rectification column 7, which is free from both impurities having higher boiling points and impurities having lower boiling points than that of oxygen, is taken out of the column bottom through a pipe P30 as an ultra-high purity liquid oxygen product, and further taken out of the gas phase above the reservoir thereof through a pipe P31 as ultra-high purity oxygen gas. This low temperature oxygen gas is introduced to the heat exchanger 3 via the pipe P31, where it is warmed to normal temperature through a counter current heat exchange with the feed air flowing thereunto from the pipe P3, and then it is taken out as an ultra-high purity oxygen gas product through a pipe P32.

[0035] Since there is a danger that hydrocarbons having higher boiling points than that of oxygen such as methane and acetylene, accumulated in the liquid oxygen stored in the column bottom of the second rectification column 5, may explode through a reaction with oxygen, a portion of the liquid oxygen is extracted from the column bottom through a pipe P37, and it is evaporated, in an auxiliary heat exchanger 10, through a counter current heat exchange with the feed air introduced therein through a pipe P35 branched from the pipe P2, and then exhausted to the open air via a pipe P38 and a pressure regulation valve V12. The air as a warming source here is cooled down, taken out through a pipe P36, joined to the pipe P8, and sent to the expansion turbine 9.

[0036] The ultra-high purity nitrogen and oxygen generator according to the present invention can give the following effects inherent in the present invention because it is constructed as mentioned above and has functions accompanied with the aforementioned construction.

[0037] In the first rectification column, ultra-high purity nitrogen free from impurities having higher boiling points and impurities having lower boiling points than that of nitrogen can be obtained by taking out liquid nitrogen from slightly below the column top portion thereof, to which the high purity liquid nitrogen is returned from the nitrogen condenser.

[0038] The oxygen-enriched liquid air separated to the column bottom of the first rectification column is rectified in the second rectification column so as to be separated to the column bottom thereof as liquid oxygen whose oxygen concentration is further increased, and to the third rectification column, this liquid oxygen is not

fed as it is, but the evaporated gas thereof is fed. Accordingly, impurities having higher boiling points than that of oxygen, contained in the liquid oxygen, are merely accompanied in a slight amount to the third rectification column. From the column top of the second rectification column, in addition, nitrogen and also impurities having lower boiling points than that of nitrogen are exhausted.

[0039] The high purity oxygen gas taken out from above the rectifying portion of the third rectification column is fed to the fourth rectification column and not liquid oxygen. Accordingly, this light purity oxygen gas is free from high boiling point impurities, and through its rectification in the fourth rectification column, ultra-high purity liquid oxygen, from which low boiling point impurities have been also removed, can be separated to the column bottom thereof.

[0040] Owing to the aforementioned construction, ultra-high purity nitrogen and ultra-high purity oxygen can be produced from one unit only by carrying out the liquefaction and rectification of feed air, without requiring another purification apparatus.

Claims

1. Process for the production of ultra-high purity nitrogen and oxygen, in which compressed feed air left after removal of impurities therefrom is cooled down for liquefaction, and introduced to a lower portion of a first rectification column (4) having a top condenser (8) wherein nitrogen from the top of the first rectification column condenses, so that through its rectification in a rectifying portion (4b, 4c, 4d) of the first rectification column, ultra-high purity nitrogen is taken out of an upper portion of the first rectification column (4), and ultra-high purity oxygen is produced at the same time, and after oxygen-enriched liquid air taken out of the lower portion of the first rectification column (4) is reduced in pressure, it is introduced to a second rectification column (5), so that through its rectification in a rectifying portion (5b) of the second rectification column, liquid oxygen is stored in a bottom portion of the second rectification column (5), the same liquid oxygen is warmed by a reboiler (5a) so as to be turned to oxygen gas containing a trace amount of impurities, the same oxygen gas is purified in a third rectification column (6), said third rectification column primarily serving to remove components in the oxygen gas, having boiling points higher than that of oxygen, therefrom by distillation, said components being removed from the bottom of the third rectification column and sent to the bottom of the second rectification column and the purified oxygen gas being thereafter introduced to a fourth rectification column (7), for removal of impurities having lower boiling points than that of oxygen, so that following rectification in a rectifying portion (7b, 7c) of the fourth rectification column, ultra-high purity oxygen is taken out from below a rectifying portion thereof and oxygen-enriched liquid air from the bottom of the first rectification column is vaporised in said top condenser.

2. Process according to claim 1 wherein part of the oxygen-enriched liquid air from the first column (4) evaporated in the top condenser (8) is used to cool the feed air prior to liquefaction in a heat exchanger (3).

3. Process according to claim 1 or 2 wherein part of the liquid oxygen stored in the second column (5) is evaporated by heat exchange with the feed air in a heat exchanger (10) so as to cool the feed air prior to liquefaction.

4. Process according to any preceding claim wherein gas is removed from the lower portion of the first column (4), sent to the reboiler (5a) of the second column (5) and is condensed therein.

5. Process according to any preceding claim wherein gas is removed from the lower portion of the first column (4), sent to a bottom reboiler (7a) of the fourth column (7) and is condensed therein.

6. Process according to any preceding claim wherein liquid nitrogen from a top condenser (8) of the first condenser (4) is sent to the top condenser (6a) of the third column (6).

7. Process according to any preceding claim wherein liquid nitrogen from a top condenser (8) of the first column (4) is sent to the top condenser (7a) of the fourth column (7).

8. An ultra-high purity nitrogen and oxygen generator comprising means for purifying and cooling compressed feed air, a first rectification column (4) for rectification of said feed air introduced into a lower portion thereof, having a rectifying portion (4b, 4c, 4d) and a top condenser (8) to produce ultra-high purity nitrogen and means for simultaneously producing ultra-high purity oxygen, said means for producing ultra-high purity oxygen comprising second, third and fourth rectification columns (5, 6, 7), means (V3) for reducing the pressure of oxygen-enriched liquid air from the lower portion of the first column (4) and introducing said reduced-pressure liquid air into the second column (5) for rectification in a rectifying portion (5b) thereof to produce and store liquid oxygen in a bottom portion of the second column (5), a reboiler (5a) for vaporising said liquid oxygen to form gaseous oxygen, means for introducing the gaseous oxygen into the third column (6)

for purification, means for introducing said purified gaseous oxygen from said third column into the fourth column (7) for rectification in a rectifying portion (7b, 7c) thereof and means for removing ultra-high purity oxygen from a region below a rectifying portion (7b, 7c) said third column (6) serving primarily to remove impurities having a higher boiling point than that of oxygen and said fourth column (7) removes impurities having a lower boiling point than that of oxygen and comprising means (P20) for removing said impurities from the bottom of the third rectification column (6) and for sending said impurities to the bottom of the second rectification column (5) and further comprising means for sending oxygen-enriched liquid air from said first rectification column to said top condenser.

9. A generator according to claim 8 wherein said third column (6) has a top condenser (6e).

10. A generator according to claim 8 or 9 wherein said fourth column (7) has a top condenser (7e).

Revendications

1. Procédé de production d'azote et d'oxygène de pureté extrêmement élevée, dans lequel de l'air d'alimentation comprimé restant après en avoir éliminé les impuretés est refroidi en vue d'une liquéfaction, et introduit dans une portion inférieure d'une première colonne de rectification (4) présentant un condenseur supérieur (8) dans lequel de l'azote provenant du haut de la première colonne de rectification se condense, de sorte que par sa rectification dans une portion de rectification (4b, 4c, 4d) de la première colonne de rectification, on fait sortir de l'azote de pureté extrêmement élevée d'une portion supérieure de la première colonne de rectification (4), et de l'oxygène de pureté extrêmement élevée est produit simultanément, et après avoir réduit en pression l'air liquide enrichi en oxygène que l'on a fait sortir de la portion inférieure de la première colonne de rectification (4), il est introduit dans une deuxième colonne de rectification (5), de sorte que par sa rectification dans une portion de rectification (5b) de la deuxième colonne de rectification, de l'oxygène liquide est stocké dans une portion du bas de la deuxième colonne de rectification (5), le même oxygène liquide est chauffé par un rebouilleur (5a) de manière à le transformer en oxygène gazeux contenant une quantité d'impuretés à l'état de traces, le même oxygène gazeux est purifié dans une troisième colonne de rectification (6), ladite troisième colonne de rectification servant principalement à éliminer des composants dans l'oxygène gazeux ayant des points d'ébullition supérieurs à celui de l'oxygène, par distillation, lesdits composants étant éliminés par le bas de la troisième colonne de rectification et envoyés au bas de la deuxième colonne de rectification, et l'oxygène gazeux purifié étant ensuite introduit dans une quatrième colonne de rectification (7), en vue d'une élimination d'impuretés ayant des points d'ébullition inférieurs à celui de l'oxygène, de sorte que suite à la rectification dans une portion de rectification (7b, 7c) de la quatrième colonne de rectification, on fait sortir de l'oxygène de pureté extrêmement élevée d'en dessous d'une portion de rectification de celle-ci et de l'air liquide enrichi en oxygène provenant du bas de la première colonne de rectification est vaporisé dans ledit condenseur supérieur.

2. Procédé selon la revendication 1, dans lequel une partie de l'air liquide enrichi en oxygène provenant de la première colonne (4), évaporée dans le condenseur supérieur (8), est utilisée pour refroidir l'air d'alimentation avant la liquéfaction dans un échangeur de chaleur (3).

3. Procédé selon la revendication 1 ou 2, dans lequel une partie de l'oxygène liquide stocké dans la deuxième colonne (5) est évaporée par échange de chaleur avec l'air d'alimentation dans un échangeur de chaleur (10) de manière à refroidir l'air d'alimentation avant la liquéfaction.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel du gaz est retiré de la portion inférieure de la première colonne (4), envoyé au rebouilleur (5a) de la deuxième colonne (5) et y est condensé.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel du gaz est retiré de la portion inférieure du premier condenseur (4), envoyé au rebouilleur inférieur (7a) de la quatrième colonne (7) et y est condensé.

6. Procédé selon l'une quelconque des revendications précédentes, dans lequel de l'azote liquide provenant d'un condenseur supérieur (8) du premier condenseur (4) est envoyé au condenseur supérieur (6a) de la troisième colonne (6).

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel de l'azote liquide provenant d'un condenseur supérieur (8) de la première colonne (4) est envoyé au condenseur supérieur (7a) de la quatrième colonne (7).

8. Générateur d'azote et d'oxygène de pureté extrêmement élevée, comprenant des moyens pour la purification et le refroidissement d'air d'alimentation comprimé, une première colonne de rectification (4) pour la rectification dudit air d'alimentation introduit

dans une portion inférieure de celle-ci, présentant une portion de rectification (4b, 4c, 4d) et un condenseur supérieur (8) pour produire de l'azote de pureté extrêmement élevée et des moyens pour la production simultanée d'oxygène de pureté extrêmement élevée, lesdits moyens pour la production d'oxygène de pureté extrêmement élevée comprenant une deuxième, une troisième et une quatrièmes colonnes de rectification (5, 6, 7), un moyen (V3) pour la réduction de la pression de l'air liquide enrichi en oxygène provenant de la portion inférieure de la première colonne (4) et l'introduction dudit air liquide sous pression réduite dans la deuxième colonne (5) pour la rectification dans une portion de rectification (5b) de celle-ci pour produire et stocker de l'oxygène liquide dans une portion du bas de la deuxième colonne (5), un rebouilleur (5a) pour la vaporisation dudit oxygène liquide pour former de l'oxygène gazeux, des moyens pour l'introduction de l'oxygène gazeux dans la troisième colonne (6) en vue d'une purification, des moyens pour l'introduction dudit oxygène gazeux purifié de ladite troisième colonne dans la quatrième colonne (7) pour la rectification dans une portion de rectification (7b, 7c) de celle-ci et des moyens pour le retrait d'oxygène de pureté extrêmement élevée d'une région en dessous d'une portion de rectification (7b, 7c), ladite troisième colonne (6) servant principalement à éliminer des impuretés ayant un point d'ébullition supérieur à celui de l'oxygène et ladite quatrième colonne (7) élimine des impuretés ayant un point d'ébullition inférieur à celui de l'oxygène, et comprenant un moyen (P20) pour l'élimination desdites impuretés par le bas de la troisième colonne de rectification (6) et pour l'envoi desdites impuretés au bas de la deuxième colonne de rectification (5), et comprenant en outre un moyen pour l'envoi d'air liquide enrichi en oxygène de ladite première colonne de rectification audit condenseur supérieur.

9. Générateur selon la revendication 8, dans lequel ladite troisième colonne (6) a un condenseur supérieur (6e).

10. Générateur selon la revendication 8 ou 9, dans lequel ladite quatrième colonne (7) a un condenseur supérieur (7e).

Patentansprüche

1. Verfahren zur Herstellung von höchstreinem Stickstoff und Sauerstoff, bei dem verdichtete Speiseluft nach Entfernen von Verunreinigungen daraus zur Verflüssigung abgekühlt wird und in einen unteren Abschnitt einer ersten Rektifizierkolonne (4) eingeleitet wird, die einen Kopfkondensator (8) hat, in dem Stickstoff aus dem Kopf der ersten Rektifizierkolonne kondensiert, so daß infolge deren Rektifikation in einem Rektifizierabschnitt (4b, 4c, 4d) der ersten Rektifizierkolonne höchstreiner Stickstoff aus einem oberen Abschnitt der ersten Rektifizierkolonne (4) entnommen wird und gleichzeitig höchstreiner Sauerstoff hergestellt wird, und, nachdem der Druck der aus dem unteren Abschnitt der ersten Rektifizierkolonne (4) entnommenen, mit Sauerstoff angereicherten, flüssigen Luft reduziert wurde, sie in eine zweite Rektifizierkolonne (5) eingeleitet wird, so daß infolge deren Rektifikation in einem Rektifizierabschnitt (5b) der zweiten Rektifizierkolonne flüssiger Sauerstoff in einem Sumpfabschnitt der zweiten Rektifizierkolonne (5) gespeichert wird, derselbe flüssige Sauerstoff mittels eines Aufkochers (5a) erwärmt wird, so daß er in Sauerstoffgas überführt wird, das eine sehr geringe Menge an Verunreinigungen enthält, welches Sauerstoffgas in einer dritten Rektifizierkolonne (6) gereinigt wird, welche hauptsächlich dazu dient, Komponenten in dem Sauerstoffgas, die höhere Siedepunkte als der Sauerstoff haben, durch Destillation daraus zu entfernen, wobei die Komponenten aus dem Sumpf der dritten Rektifizierkolonne entfernt und zum Sumpf der zweiten Rektifizierkolonne geleitet werden, und das gereinigte Sauerstoffgas danach in eine vierte Rektifizierkolonne (7) zum Entfernen von Verunreinigungen, die niedrigere Siedepunkte als der Sauerstoff haben, eingeleitet wird, so daß im Anschluß an die Rektifikation in einem Rektifizierabschnitt (7b, 7c) der vierten Rektifizierkolonne höchstreiner Sauerstoff unterhalb eines Rektifizierabschnitts derselben entnommen wird und mit Sauerstoff angereicherte flüssige Luft aus dem Sumpf der ersten Rektifizierkolonne in dem Kopfkondensator verdampft wird.

2. Verfahren nach Anspruch 1, bei dem ein Teil der mit Sauerstoff angereicherten, flüssigen Luft, die in dem Kopfkondensator (8) der ersten Kolonne (4) verdampft wurde, zum Kühlen der Speiseluft vor der Verflüssigung in einem Wärmetauscher (3) verwendet wird.

3. Verfahren nach Anspruch 1 oder 2, bei dem ein Teil des in der zweiten Kolonne (5) gespeicherten, flüssigen Sauerstoffs durch Wärmeaustausch mit der Speiseluft in einem Wärmetauscher (10) verdampft wird, so daß die Speiseluft vor der Verflüssigung gekühlt wird.

4. Verfahren nach einem der vorangehenden Ansprüche, bei dem Gas aus dem unteren Abschnitt der ersten Kolonne (4) entfernt, zu dem Aufkocher (5a) der zweiten Kolonne (5) geleitet und darin kondensiert wird.

5. Verfahren nach einem der vorangehenden Ansprü-

che, bei dem Gas aus dem unteren Abschnitt der ersten Kolonne (4) entfernt, zu einem Sumpf-Aufkocher (7a) der vierten Kolonne (7) geleitet und darin kondensiert wird.

6. Verfahren nach einem der vorangehenden Ansprüche, bei dem flüssiger Stickstoff von einem Kopfkondensator (8) der ersten Kolonne (4) zu dem Kopfkondensator (6a) der dritten Kolonne (6) geleitet wird.

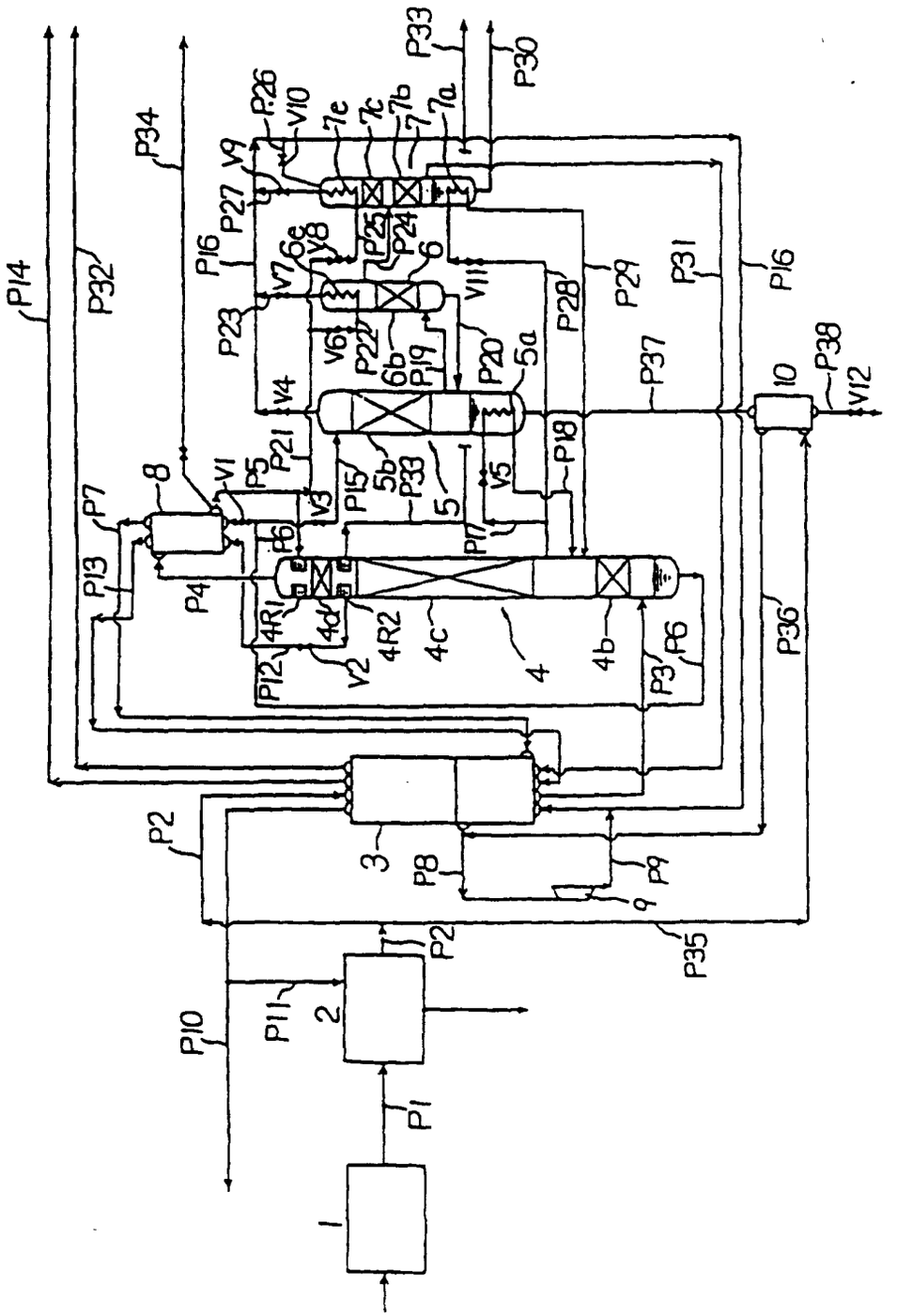
7. Verfahren nach Anspruch 6, bei dem flüssiger Stickstoff von einem Kopfkondensator (8) der ersten Kolonne (4) zu dem Kopfkondensator (7a) der vierten Kolonne (7) geleitet wird.

8. Erzeuger für höchstreinen Stickstoff und Sauerstoff, umfassend Mittel zum Reinigen und Kühlen von verdichteter Speiseluft, eine erste Rektifizierkolonne (4) zur Rektifikation der in einen unteren Abschnitt derselben eingeleiteten Speiseluft, mit einem Rektifizierabschnitt (4b, 4c, 4d) und einem Kopfkondensator (8) zur Herstellung von höchstreinem Stickstoff und Mitteln zur gleichzeitigen Herstellung von höchstreinem Sauerstoff, wobei die Mittel zur Herstellung von höchstreinem Sauerstoff eine zweite, dritte und vierte Rektifizierkolonne (5, 6, 7), Mittel (V3) zum Reduzieren des Drucks der mit Sauerstoff angereicherten, flüssigen Luft aus dem unteren Abschnitt der ersten Kolonne (4) und zum Einleiten der druckreduzierten, flüssigen Luft in die zweite Kolonne (5) zur Rektifikation in einem Rektifizierabschnitt (5b) derselben zur Herstellung und zur Speicherung von flüssigem Sauerstoff in einem Sumpfabschnitt der zweiten Kolonne (5), einen Aufkocher (5a) zum Verdampfen des flüssigen Sauerstoffs zur Bildung gasförmigen Sauerstoffs, Mittel zum Einleiten des gasförmigen Sauerstoffs in die dritte Kolonne (6) zur Reinigung, Mittel zum Einleiten des gereinigten gasförmigen Sauerstoffs aus der dritten Kolonne in die vierte Kolonne (7) zur Rektifikation in einem Rektifizierabschnitt (7b, 7c) derselben und Mittel zum Entfernen von höchstreinem Sauerstoff aus einem Bereich unterhalb eines Rektifizierabschnitts (7b, 7c) umfassen, wobei die dritte Kolonne (6) hauptsächlich dazu dient, Verunreinigungen mit einem höheren Siedepunkt als jenem des Sauerstoffs zu entfernen, und die vierte Kolonne (7) Verunreinigungen mit einem niedrigeren Siedepunkt als jenem des Sauerstoffs entfernt, und der Erzeuger Mittel (P20) zum Entfernen dieser Verunreinigungen aus dem Sumpf der dritten Rektifizierkolonne (6) und zum Einleiten der Verunreinigungen in den Sumpf der zweiten Rektifizierkolonne (5) umfaßt und außerdem Mittel zum Einleiten sauerstoffangereicherter flüssiger Luft aus der ersten Rektifizierkolonne in den Kopfkondensator umfaßt.

9. Erzeuger nach Anspruch 8, wobei die dritte Kolonne (6) einen Kopfkondensator (6e) hat.

10. Erzeuger nach Anspruch 8 oder 9, wobei die vierte Kolonne (7) einen Kopfkondensator (7e) hat.

Fig. 1


P14
P32
P34
P33
P30
P26
V10
7e
7c
7b
7a
V9
P27
P16
6e
V8
P25
P24
6
V7
V11
P23
P28
P29
P31
P16
V6
P22
6b
P19
P20
5a
P37
10
P38
V12
V4
P18
P21
V1
P5
5b
P33
5
V5
P15
V3
P7
8
P6
P17
1
P13
P4
4R1
4d
4R2
4c
4
4b
P3
P6
P36
P12
V2
3
P8
9
P9
P2
P35
P2
P10
P11
2
P1
1